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IS : 12590 - 1988

Indian Standard

SPECIFICATION FOR GLYCERINE FOR COSMETIC INDUSTRY

UDC 661·188·1 : 665·58

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 5 *July* 1989

AMENDMENT NO. 1 DECEMBER 1995
TO
IS 12590 : 1988 SPECIFICATION FOR GLYCERINE FOR COSMETIC INDUSTRY

(*Page* 1, *clause* 0.5) — Substitute the following for the existing clause:

'0.5 To check the presence of adulterants such as diethylene glycol, ethylene glycol and propylene glycol, a test method has been provided as Annex B for guidance.'

(*Page* 9, *Appendix* A) — Add the following new appendix at the end:

'APPENDIX B

THIN LAYER CHROMATOGRAPHIC ANALYSIS FOR DETECTION OF DIETHYLENE GLYCOL, ETHYLENE GLYCOL AND PROPYLENE GLYCOL AS ADULTERANTS IN GLYCERINE

(*Clause* 0.5)

B-0 GENERAL

The presence of diethylene glycol, ethylene glycol and propylene glycol is determined by thin layer chromatographic analysis.

B-1 PROCEDURE

B-1.1 Reagents

B-1.1.1 *Acidic Potassium Dichromate Solution*

Dissolve 7 g potassium dichromate in 70 ml water, add 10 ml of sulphuric acid slowly. Cool, mix and make up to 100 ml with water. If the solution turns green, it should be discarded.

B-1.2 Method of Analysis

A typical condition for TLC analysis of glycerine is given below:

Test solution of sample : 2% *m/v* in alcohol glycerine — A

Test solution of standard : 2% *m/v* in alcohol glycerine — B

Test solution of standard : 0.2% *m/v* in alcohol diethylene glycol — C

Test solution of standard : 0.2% *m/v* in alcohol ethylene glycol — D

Test solution of standard : 0.2% *m/v* in alcohol propylene glycol — E

Spot : 5 µl of Test solution of sample — A ,
Standard substance — B,
Standard substance — C,
Standard substance — D, and
Standard substance — E.

Thin layer plate : Silica gel

Mobile Phase : A mixture of 55 percent chloroform, 35 percent acetone and 10 percent alcohol

Run : 10 cm.

Develop the chromatogram until the solvent front has ascended about 10 cm. After removal of the plate, dry it in a current of air, heat at 105°C for five minutes. Cool and spray with acidic potassium dichromate solution. Allow to dry in air for about 15 minutes.

B-3 REPORT

The blue principal spot in the chromatogram obtained with solution A shall correspond to the spot obtained with solution B and there shall be no other additional spot other than the principal spot obtained with solution A corresponding to the spots obtained with solutions C, D and E.'

(PCD 19)

Indian Standard
SPECIFICATION FOR GLYCERINE FOR COSMETIC INDUSTRY

0. FOREWORD

1 This Indian Standard was adopted by the ureau of Indian Standards on 23 December 988, after the draft finalized by the Cosmetics ectional Committee had been approved by the etroleum, Coal and Related Products Division ouncil.

.2 Glycerine is derived from naturally occurng products, such as, oils and fats, and also om soap industry as a by-product. It is a ery important raw material for cosmetic dustry and is mainly used in formulation of entifrices, soaps, skin creams and hair care roducts as a humectant.

.3 Glycerine is capable of maintaining acteriological stability in aqueous systems, ossesses good flavour and its safety on ingestion as been well established by specific laboratory nd clinical trials and human use experience. is capable of operating in concert with gum abilizers and may be used in products where anslucency or transparency is required.

4 Besides cosmetic industry, glycerine is also ed as a raw material for several other industries. A separate standard for glycerine covering crude as well as refined grades (*see* IS : 1796-1986*) has also been published. The present standard has been drawn up specifically to cater to the needs of cosmetic industry.

0.5 The Sectional Committee felt the need of stipulating a requirement to check the presence of toxic adulterants, such as, glycols in glycerine. In the absence of standardized test procedure and data thereon, the same would be included at a later date when detailed information is avilable in this regard.

0.6 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960†. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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*Specification for glycerine (*second revision*).

†Rules for rounding off numerical values (*revised*).

SCOPE

1 This standard prescribes requirements and ethods of sampling and test for glycerine for smetic industry.

REQUIREMENTS

1 **Description** — The material shall be a ear, colourless, odourless, syrupy liquid, conting essentially of glycerol. It shall have a eet taste followed by a sensation of warmth. shall be miscible in all proportions with water d with ethyl alcohol (90 percent *v/v*) and it all not be soluble in diethyl ether, chloroform fixed oils. It shall be free from visible eign matter.

2 The material shall comply with the requireents given in Table 1.

PACKING AND MARKING

Packing — The material shall be supplied suitable well closed containers as agreed to tween the purchaser and the supplier.

3.2 Marking — The containers shall be securely closed and legibly marked with the following information:

a) Manufacturer's name;
b) Recognized trade-mark, if any;
c) Name, type and grade of the material;
d) Mass of the material in the container;
e) Month and year of manufacture; and
f) Batch number in code or otherwise to enable the lot of manufacture to be traced back from records.

3.1.1 The containers may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard

TABLE 1 REQUIREMENTS FOR GLYCERINE

(*Clauses* 2.2 *and* 5.1)

Sl No.	Characteristic	Requirement	Method of Test, Ref to Cl No. in Appendix A
(1)	(2)	(3)	(4)
i)	Glycerol, percent by mass, *Min*	98·0	A-2
ii)	Relative density at 30/30°C, *Min*	1·255 2	A-3
iii)	Colour reading on Lovibond scale expressed as $Y + R$ not deeper than (in 5¼ inch cell)	1·0	A-4
iv)	Ash, percent by mass, *Max*	0·01	A-5
v)	Alkalintiy (as Na_2O), percent by mass, *Max*	0·01	A-6
vi)	Arsenic (as As), parts per million, *Max*	2	A-7
vii)	Copper	To pass the test	A-8
viii)	Iron (as Fe), parts per million, *Max*	2	A-9
ix)	Lead (as Pb), parts per million, *Max*	5	A-10
x)	Chlorides (as Cl), parts per million, *Max*	10	A-11
xi)	Sulphates (as SO_4), parts per million, *Max*	10	A-12
xii)	Fatty acids and esters (as Na_2O), percent by mass, *Max*	0·06	A-13
xiii)	Acraldehyde and glucose	To pass the test	A-14

marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in IS : 3958-1984*.

5. TEST METHODS

5.1 Unless specified otherwise, tests shall be carried out as prescribed in Appendix A. Reference to the relevant clauses of Appendix A is given in col 4 of Table 1.

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*Methods of sampling cosmetics (*first revision*).

APPENDIX A

(*Clause* 5.1, and *Table* 1)

METHODS OF TEST FOR GLYCERINE

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

Note — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF GLYCEROL CONTENT

A-2.0 Outline of the Method — Glycerine reacts with sodium metaperiodate ($NaIO_4$) in acid aqueous solution to produce formaldehyde and formic acid; the latter is used as a measure of glycerol. Other polyhydric alcohols with three or more adjacent hydroxyl groups also produce formic acid and, therefore, interfere in glycerol determination. The method cannot,

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*Specification for water for general laboratory use (*second revision*).

therefore, be used directly on samples containing sugars.

A-2.1 Apparatus

A-2.1.1 *Burette* — 50 ml capacity, with 0·05 ml graduations.

A-2.1.2 *Pipette* — 50 ml capacity.

A-2.1.3 *Conical Flasks* — 500 ml capacity, with glass stopper.

A-2.1.4 *p*H *Meter* — with glass electrode.

A-2.1.5 *Weighing Pipette or Bottle*

A-2.2 Reagents

A-2.2.1 *Standard Sodium Hydroxide Solution* — 0·1 N, accurately standardized. The solution shall be carbonate-free and should be cross-checked periodically against standard glycerol (*see* **A-2.2.7**) by the method described in **A-2.3**.

A-2.2.2 *Sodium Hydroxide Solution* — 0·05 N.

A-2.2.3 *Phenol Red Indicator Solution* — 0·05 percent (*m/v*) solution. Dissolve 0·05 g of phenol red in 10 ml of ethyl alcohol and make up to 100 ml with water.

A-2.2.4 *Dilute Sulphuric Acid* -- 0·2 N.

A-2.2.5 *Sodium Metaperiodate Solution* — Disslove 70 g of sodium metaperiodate in one litre of water containing 10 ml of approximately 1 N sulphuric acid and store the solution in an amber coloured bottle in the dark.

NOTE — Sodium metaperiodate shall be a white crystalline powder containing not less than 98 percent sodium metaperiodate. If metaperiodate does not dissolve, it is not of reagent quality and a new supply should be obtained.

A-2.2.6 *Ethylene Glycol* — neutral and free from glycerol.

A-2.2.7 *Standard Glycerol* — An aqueous solution containing about 90 percent of chemically pure glycerine, concentration of which is known from density at 30°C (*see* Table 2) and moisture by [Karl Fischer method (*see* IS : 2362-1973*)] determinations.

NOTE — If necessary, density of glycerine may be calculated by multiplying its relative density (*see* **A-3**) by the density of water at the desired temperature.

A-2.3 Procedure — Using a weighing pipette or bottle, transfer into a conical flask a well-mixed and accurately weighed sample (*see* Note 1). Add 100 ml of carbon dioxide-free water and 3 drops of phenol red indicator solution, and acidify with sulphuric acid solution to a definite yellow colour. Heat to boiling and cool to room temperature. Adjust the *p*H value to about 8·0 to 8·1 by adding sodium hydroxide solution (*see* **A-2.2.2**) drop by drop until the colour changes from yellow to just pink. If the colour of the original solution interferes with the detection of the colour change of the indicator, use a *p*H meter. Pipette accurately 50 ml of sodium metaperiodate solution, replace the glass stopper, swirl for intimate mixing, and allow the flask to stand in the dark for 30 minutes. Wash down the sides of the flask with water, add 5 ml of ethylene glycol, replace the stopper, swirl gently, and allow the flask to stand in the dark for further 20 minutes. Titrate the liberated formic acid with standard sodium hydroxide solution (*see* **A-2.2.1**) from yellow to pink end point. Carry out a blank test simultaneously under similar test conditions (*see* Note 2).

NOTE 1 — The mass in grams of the sample should be within the limits given by the formula $(41 \pm 9)/P$, where P is the expected percentage of glycerol in the sample. When the glycerol content is not known, weigh the amount specified for 100 percent glycerol and from the results of this test, select the proper size sample considering that the results will tend to be high if the sample weighed is smaller than that required for the most accurate results.

NOTE 2 - If a *p*H meter is used, the end point for the blank should be *p*H 6·5 ± 0·1 and that for the sample 8·1 ± 0·1.

A-2.4 Calculation

$$\text{Glycerol content, percent by mass} = \frac{9{\cdot}209\,(S - B)\,N}{M}$$

where

S - volume in ml of standard sodium hydroxide solution required for the sample;

B = volume in ml of standard sodium hydroxide solution required for the blank;

N = normality of standard sodium hydroxide solution; and

M = mass in g of the material taken for the test.

NOTE — When the titration is carried out with phenol red solution as the indicator, add 0·3 percent (expressed on 100 percent glycerol) to the observed glycerol result. No correction is necessary if a *p*H meter is used.

A-3. DETERMINATION OF RELATIVE DENSITY

A-3.0 General — The relative density of glycerine shall be expressed as the ratio of the mass in air of a given volume of the material at 30°C to that of an equal volume of water at the same temperature. The relative density may be determined with a hydrostatic balance or relative density bottle. The relative density bottle method shall be adopted as the referee method in cases of dispute.

---

*Determination of water by the Karl Fischer method (*first revision*).

TABLE 2 GLYCEROL CONTENT AND DENSITY OF AQUEOUS SOLUTIONS OF REFINED GLYCERINE*

(*Clause* A-2.2.7)

Glycerol	Density at 30°C	Glycerol	Density at 30°C
(1)	(2)	(1)	(2)
percent	g/ml	percent	g/ml
100	1·254 95	75	1·189 00
99	1·252 35	74	1·186 35
98	1·249 75	73	1·183 65
97	1·247 10	72	1·181 00
96	1·244 50	71	1·178 30
95	1·241 90	70	1·175 65
94	1·239 30	69	1·172 90
93	1·236 70	68	1·170 20
92	1·234 10	67	1·167 45
91	1·231 50	66	1·164 70
90	1·228 90	65	1·161 95
89	1·226 25	64	1·159 25
88	1·223 60	63	1·156 50
87	1·220 95	62	1·153 75
86	1·218 30	61	1·151 00
85	1·215 65	60	1·148 30
84	1·213 00	59	1·145 55
83	1·210 35	58	1·142 85
82	1·207 70	57	1·140 10
81	1·205 05	56	1·137 40
80	1·202 40	55	1·134 70
79	1·199 70	54	1·131 96
78	1·197 05	53	1·129 25
77	1·194 35	52	1·126 50
76	1·191 70	51	1·123 80
		50	1·121 10

*Reproduced from Bosart and Sncddy. Industrial and Enginnering Chemistry, Ed 20, 1378 (1928).

A-3.1 Hydrostatic Balance Method

A-3.1.0 *General* — There are several forms of hydrostatic balances and the procedure described below is applicable to a chain analytical balance.

A-3.1.1 *Procedure* — Take a sufficient quantity of freshly boiled and cooled water at 30°C in a cylinder (about 125 mm high and 25 mm inside diameter), and place it on the adjustable platform inside the balance. Suspend clean dry plummet in the cyliner and adjust the platform so that the loop of the wire by which the plummet is suspended just dips in water. Place the rider on the beam in such a position that the pointer swings equally on either side of zero on the scale. Make the final adjustment using the vernier scale and note the reading. Lower the platform and remove the plummet and the cylinder. Wipe the plummet dry. Fill another cylinder with the test sample at 30°C and repeat the above procedure ensuring that no air bubbles are formed in the eye hole of the plummet. Take the reading as described previously for water. As glycerine is very hygroscopic, it is essential that the test is performed as rapidly as possible.

A-3.1.2 *Calculation*

$$\text{Apparent relative density at } 30/30°\text{C} = \frac{A}{B}$$

where

A = scale reading with the material taken for the test at 30°C, and

B = scale reading with water at 30°C.

A-3.2 Relative Density Bottle Method

A-3.2.1 *Apparatus*

A-3.2.1.1 *Relative density bottle* — A relative density bottle of 50-ml capacity with central neck fitted with ground-glass thermometer which serves as a stopper, graduated from 10 to 40°C in 0·2°C divisions and an overflow side tube with ground in glass cap, as shown in Fig. 1.

To calibrate, clean and dry the bottle thoroughly, weigh and then fill with freshly and cooled water at about 25°C after removing the cap of the side arm. Filling should be done to overflowing by holding the bottle on its side.

Insert the stopper in such a way as to prevent the entrapment of air bubbles. Excess water overflows through the side tube. Immerse in a water bath maintained at 30·0 ± 0·2°C. Keep the entire bulb completely covered with water. Allow the temperature to rise until the temperature inside the bottle and that of the bath are the same. This takes about 30 minutes. Carefully remove any water which has exuded from the side opening and replace the cap on the side tube. Remove the bottle from the bath and wipe completely dry equilibriate to room temperature and weigh. Calculate the mass of water. This is a content for the bottle but should be checked periodically.

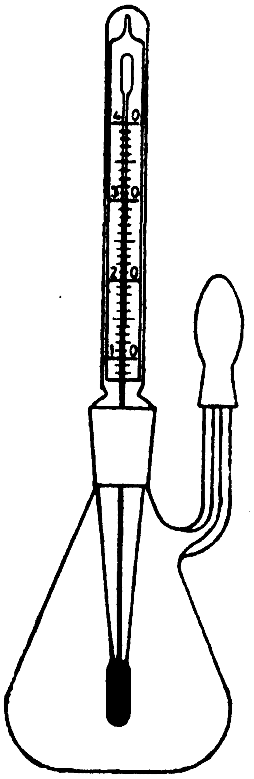

Fig. 1 Relative Density Bottle

A-3.2.1.2 *Water bath* — maintained at 30·0 ± 0·2°C.

A-3.2.1.3 *Thermometer* — Any convenient thermometer of a suitable range with 0·1 or 0·2°C sub-divisions. The thermometer should be checked against a standard thermometer which has been calibrated and certified by the National Physical Laboratory, New Delhi or any other laboratory recognized by the Goverment of India for such work.

A-3.2.2 *Procedure* — Fill the clean and dry bottle with the test sample previously cooled to about 25°C, after removing the cap of the side arm, holding the bottle on its side in such a manner as to prevent entrapment of air bubbles. Insert the stopper in such a way that the capillary of the side tube is completely filled with glycerine. Immerse and hold the bottle in the water bath at 30 ± 0·2°C so that the entire bulb is covered with water. Allow the temperature to rise until the temperatures of the water bath and that of the glycerine in the relative density bottle are the same. This takes about 30 minutes. Carefully wipe off any glycerine that has come through the capillary opening and replace the cap on the side tube. Remove the bottle from the bath, clean and dry the outside thoroughly. Equilibriate to room temperature and weigh.

A-3.2.2.1 It is advisable to carry out the test in duplicate.

A-3.2.3 *Calculation*

$$\text{Relative density at } 30/30^\circ\text{C} = \frac{A - B}{C - B}$$

where

A = mass in g of the relative density bottle with the glycerine taken for the test at 30°C,

B = mass in g of the relative density bottle, and

C = mass in g of the relative density bottle with water at 30°C.

A-3.3 Corrections — Sometimes, specially when using the hydrostatic balance, it is not convenient to make the determination at the specified temperature, namely, 30°C. The determination may be made at a convenient temperature T as near to 30°C as possible and the result corrected to 30°C as follows:

$$\text{Relative density at } 30/30^\circ\text{C} = \frac{ab + Kc\,(T - 30)}{c}$$

where

a = relative density of glycerine at T/T°C;

b = density of water at T°C;

c = density of water at 30°C, namely, 0·995 67;

T = temperature at which relative density was determined; and

K = change in relative density per degree celsius. The value for K shall be selected as follows:

Transposition	K
From 15°/15°C to 20°/20°C and vice versa	0·000 40
„ 20°/20°C „ 25°/25°C „ „ „	0·000 32
„ 25°/25°C „ 30°/30°C „ „ „	0·000 27
„ 30°/30°C „ 35°/35°C „ „ „	0·000 23

A-4. DETERMINATION OF COLOUR

A-4.0 General — This method determines the colour of glycerine by comparison with Lovibond glasses of known colour characteristics.

A-4.1 Apparatus

A-4.1.1 *Lovibond Tintometer*

A-4.1.2 *Glass Cells* — 5¼ inch cell. The cell should be of clear, colourless glass.

A-4.2 Procedure — Select a clean dry glass cell of the prescribed size and fill it with the well-mixed sample. Put the cell in position in the tintometer and place along side of it such yellow, red and/or neutral Lovibond glass slides as are necessary to match the colour shade of the glycerine, observing the colours of the glycerine and of the combination of the glass slides through an eye piece.

A-4.3 Report the colour of the material under test in terms of Lovibond units as follows:

$$\text{Colour reading in } 5\tfrac{1}{4} \text{ inch cell} = Y + R$$

where

Y = the sum total of the various yellow slides used, and

R = the sum total of the various red slides used.

A-5. DETERMINATION OF ASH

A-5.1 Apparatus

A-5.1.1 *Platinum Dish or Crucible*

A-5.1.2 *Desiccator* — containing concentrated sulphuric acid.

A-5.2 Procedure — Weigh accurately about 50 g of the sample in a platinum dish and heat it gently to a point where glycerine vapours are evolved and can be ignited. Ignite the glycerine, remove the burner, and allow the glycerine to burn down to a brown or black residue. Cool for 1 minute, recommence heating and incinerate the residue at not more than dull red heat (550°C *Max*) to a white or grey white ash. Cool the dish in a desiccator and weigh the residue.

A-5.3 Calculation

$$\text{Ash, percent by mass} = \frac{100\,m}{M}$$

where

m = mass in g of the residue, and

M = mass in g of the material taken for the test.

A-6. DETERMINATION OF ALKALINITY

A-6.0 General — Alkalinity represents the free titrable alkali present in samples of refined glycerine.

A-6.1 Apparatus

A-6.1.1 *Conical Flask* – 500 ml capacity.

A-6.2 Reagents

A-6.2.1 *Standard Hydrochloric Acid* – 0·1 N, accurately standardized.

A-6.2.2 *Phenolphthalein Indicator Solution* — 1 percent ethyl alcohol (*m*/*v*) in 95 percent ethyl alcohol (*v*/*v*) or rectified spirit (conforming to IS : 323-1959*).

A-6.3 Procedure — Weigh accurately about 100 g of the well-mixed sample into a 500-ml conical flask and add 150 ml of carbon dioxide-free water. Titrate with standard solution of hydrochloric acid using phenolphthalein solution as indicator.

A-6.4 Calculation

$$\text{Alkalinity (as } Na_2O \text{) percent by mass} = \frac{3{\cdot}1\,AN}{M}$$

where

A = volume in ml of standard hydrochloric acid used,

N = normality of standard hydrochloric acid, and

M = mass in g of the material taken for the test.

A-7. DETERMINATION OF ARSENIC

A-7.1 Procedure — Weigh accurately about 10 g of the well-mixed sample and carry out the determination as prescribed IS : 2088-1983†.

A-8. TEST FOR COPPER

A-8.1 Reagents – The following reagents are required and shall be free from copper impurity.

A-8.1.1 *Hydrochloric Acid* — 10 percent (*v*/*v*) solution of hydrochloric acid (rd 1·18).

A-8.1.2 *Hydrogen Sulphide Solution* — aqueous, freshly prepared.

A-8.2 Procedure — Take 10 ml of the sample in a test tube and add 30 ml of water, 1 ml of hydrochloric acid and 10 ml of hydrogen

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*Specification for rectified spirit (*revised*).

†Methods for determination of arsenic (*second revision*).

sulphide solution. The non-development of any colour indicates the absence of copper.

A-9. DETERMINATION OF IRON

A-9.1 Apparatus

A-9.1.1 *Silica Dish*

A-9.1.2 *Nessler Cylinders* — 50 ml capacity.

A-9.1.3 *Pipette* — 1 ml capacity with 0·01 ml graduations.

A-9.2 Reagents — The following reagents are required and with the exception of **A-9.2.5**, they shall be free from iron impurity.

A-9.2.1 *Potassium Hydrogen Sulphate* — fused.

A-9.2.2 *Hydrochloric Acid* — 10 percent (*v/v*) solution of hydrochloric acid (rd 1·18).

A-9.2.3 *Nitric Acid* — 10 percent (*v/v*) solution of nitric acid (rd 1·42).

A-9.2.4 *Thioglycollic Acid Solution* — 10 percent solution. Dilute 10 ml of 90 percent thioglycollic acid to 100 ml with water.

NOTE — Care shall be taken as not to allow concentrated thioglycollic acid to come in contact with the skin as it causes painful wounds which take a long time to heal.

A-9.2.5 *Standard Iron Solution* — Dissolve 7·03 g of ferrous ammonium sulphate [$FeSO_4.(NH_4)_2SO_4.6H_2O$] in water containing 5 ml of dilute sulphuric acid (1 : 3) and make up to 1 litre in a volumetric flask. Dilute this stock solution 100 times immediately before use. One millilitre of this solution contains 0·000 01 g of Fe.

A-9.2.6 *Amonia Solution* — 50 percent (*v/v*) aqueous solution, prepared from liquor ammonia (rd 0·90).

A-9.2.7 *Citric Acid Solution* — 50 percent (*m/v*), aqueous.

A-9.3 Procedure

A-9.3.1 Weigh accurately about 10 g of the sample in a silica dish and proceed to ash as described in **A-5.2**. After ashing is complete, add 0·5 g of potassium hydrogen sulphate and fuse gently until a clear melt is obtained. Allow to cool, add 5 ml of hydrochloric acid and warm, flowing the acid round the sides of the dish. Add 5 ml of nitric acid and bring to incipient boiling. Transfer the solution quantitatively to a 100-ml of volumetric flask and make up to volume with water.

A-9.3.2 Pipette out in a Nessler cylinder 25 ml of the solution obtained in **A-9.3.1**. Add 4 ml of citric acid solution add 10 ml of ammonia solution. Cool and add 5 ml of thioglycollic acid solution. Mix well after each addition. Dilute to 50 ml with distilled water. A pale purple colour is produced if iron is present.

A-9.3.3 To a second Nessler cylinder, add 0·125 g of potassium hydrogen sulphate, 25 ml of distilled water, 1·25 ml of nitric acid and 2·5 ml of hydrochloric acid. Add 4 ml of citric acid solution and 10 ml of ammonia solution. Cool and add 5 ml of thioglycollic acid solution. Mix well after each addition. Dilute to 50 ml with distilled water. From a pipette, gradually add standard iron solution so that after shaking the cylinder, the intensity of the colour matches with that produced by the material under test. Note the volume of standard iron solution used.

A-9.4 Calculation

$$\text{Iron (as Fe), parts per million} = \frac{40\,V}{M}$$

where

V = volume in ml of standard iron solution used, and

M = mass in g of the material taken for the test.

A-10. DETERMINATION OF LEAD

A-10.1 Apparatus

A-10.1.1 *Nessler Cylinders* — 50 ml capacity.

A-10.2 Reagents — The following reagents are required and, with the exception of **A-10.2.1**, they shall be free from lead impurity.

A-10.2.1 *Standard Lead Solution* — Dissolve 1·600 g of lead nitrate in 100-ml of water to which 5 ml of nitric acid has been added. Make the volume to one litre dilute 10 ml of this solution again to one litre. One millilitre of this solution is equivalent to 0·01 mg of lead as Pb.

A-10.2.2 *Potassium Cyanide Solution* — 10 percent (*m/v*). Dissolve 10 g of potassium cyanide in 90 ml of water, add 2 ml of hydrogen peroxide solution (6 percent), allow to stand for 24 hours and make up to 100 ml with water.

CAUTION — Potassium cyanide is highly poisonous chemical. It may be used with great care and shall never be used without using gloves.

A-10.2.3 *Sodium Sulphide Solution* — 10 percent (*m/v*), aqueous. Filter, if necessary.

A-10.2.4 *Ammonia Solution* — 10 percent (*m/v*). Dilute 1 part of amonia (rd 0·90) with 2·5 parts of water.

A-10.2.5 *Acetic Acid Solution* — 1 : 2 solution of glacial acetic acid in water.

A-10.3 Procedure

A-10.3.1 Prepare solutions from the sample by dissolving 14·5, 12·0, 9·5, 7·0, 4·5 and 2·0 g, respectively, in about 10 ml of hot water containing 5 ml of acetic acid solution. To the solution containing 2·0 g of the sample, add 5 ml of standard lead solution; this will serve as the control. Make all the solutions alkaline with ammonia solution and add to each, 1 ml of potassium cyanide solution and make up to 40 ml with water. If the solutions are turbid, filter them through a filter paper. Equalize any differences in colour by adding a few drops of a highly diluted solution of burnt sugar.

A-10.3.2 Dilute all the solutions, including the control, to 50 ml with water; to each, add 2 drops of sodium sulphide solution, mix thoroughly and transfer to Nessler cylinders. Compare the colours by viewing the cylinders against a white tile. Note which one of the five test samples has a colour most closely matching with that of the control. Repeat the test using an appropriate sample size and note the amount of glycerine required to produce a colour with an intensity exactly matching that of the control.

A-10.4 Calculation

$$\text{Lead (as Pb), parts per million} = \frac{10}{M - 2}$$

where

M = mass in g of the material required to match the colour of the control sample.

A-11. DETERMINATION OF CHLORIDES

A-11.1 Nephelometric Method

A-11.1.1 *Apparatus*

A-11.1.1.1 *Nessler cylinders* — 50 ml capacity.

A-11.1.1.2 *Pipette* — 1 ml capacity, with 0·01 ml graduations.

A-11.1.2 *Reagents* — The following reagents required and, with the exception of **A-11.1.2.3**, they shall be free from chloride impurity.

A-11.1.2.1 *Nitric acid* — 35 percent (*v/v*) solution of nitric acid (rd 1·42) in water.

A-11.1.2.2 *Silver nitrate solution* — 5 percent (*m/v*), aqueous.

A-11.1.2.3 *Standard chloride solution* — Dissolve 1·647 9 g of sodium chloride in water and make up to 1 litre in volumetric flask. Dilute this stock solution 100 times immediately before use. One millilitre of this solution contains 0·000 01 g of Cl.

A-11.1.3 *Procedure*

A-11.1.3.1 Weigh accurately about 15 g of the sample in a 100 ml volumetric flask and make up to volume with water. Pipette out 25 ml of the solution in a Nessler cylinder, add 1 ml of nitric acid and 10 ml of silver nitrate solution. Make up to 50 ml with water. A dull white turbidity is produced if chlorides are present.

A-11.1.3.2 To a second Nessler cylinder, add 10 ml of silver nitrate solution and 1 ml of nitric acid and make up to 50 ml with water. From a pipette, gradually add standard chloride solution drop by drop so that after shaking the cylinder, the turbidity matches with that produced by the material under test. Note the volume of the standard chloride solution used.

A-11.1.4 *Calculation*

$$\text{Chlorides (as Cl), parts per million} = \frac{40\,V}{M}$$

where

V = volume in ml of standard chloride solution used, and

M = mass in g of the material taken for the test.

A-12. DETERMINATION OF SULPHATES

A-12.1 Apparatus

A-12.1.1 *Nessler Cylinders* — 25 ml capacity.

A-12.1.2 *Pipette* — 1 ml capacity, with 0·01 ml graduations.

A-12.2 Reagents — The following reagents are required and, with the exception of **A-12.2.3**, they shall be free from sulphate impurity.

A-12.2.1 *Hydrochloric Acid* — 1:1 solution of hydrochloric acid (rd 1·18) in water.

A-12.2.2 *Barium Chloride Solution* — 10 percent (*m/v*), aqueous.

A-12.2.3 *Standard Sulphate Solution* — Dissolve 1·479 g of anhydrous sodium sulphate in water and make up to 1 litre in a volumetric flask. Dilute this stock solution 10 times immediately before use. One millilitre of this solution contains 0·000 1 g of SO_4.

A-12.3 Procedure

A-12.3.1 Weigh accurately about 10 g of the sample into a Nessler cylinder, add 1 ml of hydrochloric acid and 2 ml of barium chloride solution, and make up to 25 ml with water. Allow to stand for 30 minutes. A dull white turbidity is produced if sulphates are present.

A-12.3.2 Simultaneously, introduce 0·5, 0·75, 1·0 and 12·5 ml of standard sulphate solution into 4 separate Nessler cylinders. In each, add 1 ml of hydrochloric acid and 2 ml of barium chloride solution, and make up to 25 ml with water. Allow to stand for 30 minutes. Note which one of these four solutions has a turbidity most closely matching with that produced by the test material (*see* **A-12.3.1**). Repeat the test and note the volume of standard sulphate solution required to produce a turbidity exactly matching with that of the test material.

A-12.4 Calculation

$$\text{Sulphates (as } SO_4\text{), parts per million} = \frac{100\,V}{M}$$

where

V = volume in ml of standard sulphate solution used, and

M = mass in g of material taken for the test.

A-13. DETERMINATION OF FATTY ACIDS AND ESTERS

A-13.0 General — Fatty acids and esters represent the free fatty acids and the fatty acid equivalent of all the esters present in the sample.

A-13.1 Apparatus

A-13.1.1 *Round-Bottomed Flask* — 500 ml capacity.

A-13.2 Reagents

A-13.2.1 *Standard Sodium Hydroxide Solution* — 0·25 N, accurately standardized.

A-13.2.2 *Standard Sulphuric Acid* — 0·25 N, accurately standardized.

A-13.2.3 *Phenolphthalein Indicator Solution* — 1 percent (*m/v*), in 95 percent ethyl alcohol (*v/v*) or rectified spirit conforming to IS : 323-1959*.

A-13.3 Procedure — Weigh accurately about 50 g of the sample into a 500-ml round-bottomed flask. Add 100 ml of hot, carbon dioxide free water and 1 ml of phenolphthalein indicator solution. If the solution is alkaline, neutralize it with sulphuric acid. Add exactly 15·0 ml of standard sodium hydroxide solution, connect the flask to a reflux condenser and heat to boiling. Boil for 5 minutes, allow to cool slightly and wash down the condenser with a little water. Disconnect the flask, close it with a stopper carrying a soda lime tube, and cool. Titrate with standard sulphuric acid. Carry out a blank test simultaneously under similar test conditions but using 140 ml of water.

A-13.4 Calculation

$$\text{Fatty acids and esters, as } Na_2O\text{, percent by mass} = \frac{3{\cdot}1\,(B - S)\,N}{M}$$

where

B = volume in ml of standard sulphuric acid required for the blank,

S = volume in ml of standard sulphuric acid required for the material,

N = normality of standard sulphuric acid, and

M = mass in g of the material taken for the test.

A-14. TEST FOR THE PRESENCE OF ACRALDEHYDE AND GLUCOSE

A-14.1 Procedure — Take about 5 ml of the sample in an evaporating dish and heat strongly. Heat further until it volatilizes and burns. The development of not more than a faint yellow, but not pink colour in the preliminary heating, and the burning of the sample with little or no charring and with no odour of burnt sugar in the subsequent heating, indicates the absence of acraldehyde and glucose.

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*Specification for rectified spirit (*revised*).

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